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**POWER AND THERMAL
TECHNOLOGY FOR AIR AND
SPACE**

**Delivery Order 0006: Nano-filled Polymers
for Electrical Insulation**



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14. ABSTRACT The objective of this delivery order was to conduct research aimed at developing nano-filled polymers for electrical insulation. Improved polymers could be used in wiring, cabling, potting compounds, and capacitors. Specifically, this effort involved research in areas such as nano-filler dispersion, bulk sample fabrication, and thin film processing. These activities are designed to contribute to the improvement of highly stressed electrical insulation with a goal of improved mean time before failure. This program focused exclusively on the addition of Polyhedral Oligomeric Silsesquioxane (POSS) nanoparticles into epoxy resin. A procedure was developed to incorporate POSS nanoparticles into a common epoxy/amine resin system at loadings over 5 wt%, where the solubility limit seems to be between 5 and 10 wt%. Dispersion of the particles was excellent in regards to optical clarity.					
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1. Introduction

The objective of this delivery order is to conduct research aimed at developing nano-filled polymers for electrical insulation. Improved polymers could be used in wiring, cabling, potting compounds, and capacitors. Improvements are needed in electrical, thermal and mechanical properties for the enhanced performance of electrical insulation. Specifically, this effort will involve research in areas such as nano-filler dispersion, bulk sample fabrication, and thin film processing. These activities are designed to contribute to the improvement of highly stressed electrical insulation with a goal of improved mean time before failure.

In this program we focused exclusively on the addition of Polyhedral Oligomeric Silsesquioxane (POSS) nanoparticles into epoxy resin. Researchers have synthesized a wide variety of POSS nanoparticle structures and investigated their incorporation into several thermoset and thermoplastic polymer matrices [1-11]. The surface of the POSS structure is often modified with reactive or otherwise “functional” groups to foster improved compatibility and/or covalent bonding with the host matrix. The most common properties studied to date include glass transition temperature (T_g), dynamic mechanical properties such as storage modulus and $\tan \delta$, and thermooxidative stability and thermogravimetric properties. The degree of improvement in these properties has varied from none to modest in the cited studies. A key material and processing issue, the quality of nanoparticle dispersion, was often poorly characterized, or not characterized at all in these same studies. Nothing was found in the literature on the issue of electrical insulation property improvement via *POSS* nanocomposites. However, studies with nano-sized Al_2O_3 , TiO_2 , and SiO_2 particles showed significant improvements in electrical breakdown strength [12-14].

2. Experimental Method

2.1. Materials

Silsesquioxanes are a class of compounds well known for their high temperature stability [4]. Recently a series of Polyhedral Oligomeric Silsesquioxanes (POSS) has been developed [15], all of which involve a robust silicon-oxygen cage framework with dimension of about 1-3 nm. A variety of organic groups can be grafted to the cage in order to “functionalize” the particle for improved interaction with the host polymer matrix. In the current program, the polymer resin system was comprised of EPON 828 (DiGlycidyl Ether of Bisphenol A) and Jeffamine D-400 (polyoxypropylene), see Figure 1. Both components are colorless liquids at room temperature, with a viscosity of 14,000 cP for EPON 828 and 20 cP for Jeffamine D-400. The stoichiometric mix ratio (not including any possible contribution from POSS) is 100 parts by weight (pbw) EPON 828 to 56 pbw Jeffamine D-400. This resin systems gels at room temperature over the course of several hours. A thermal post cure cycle is used to complete the cure.

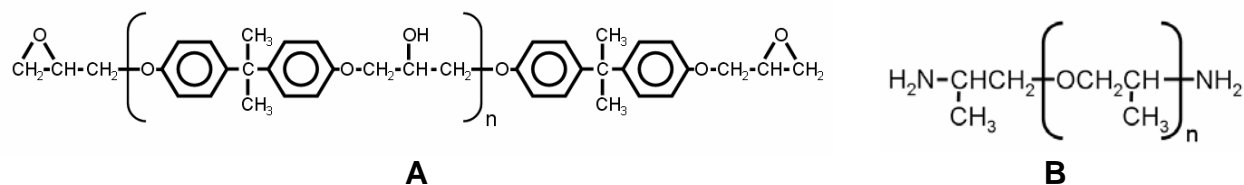
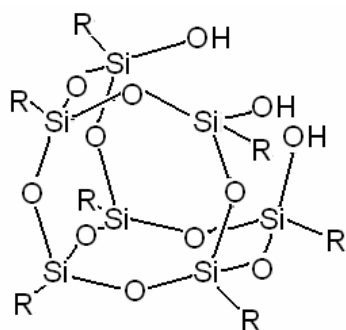


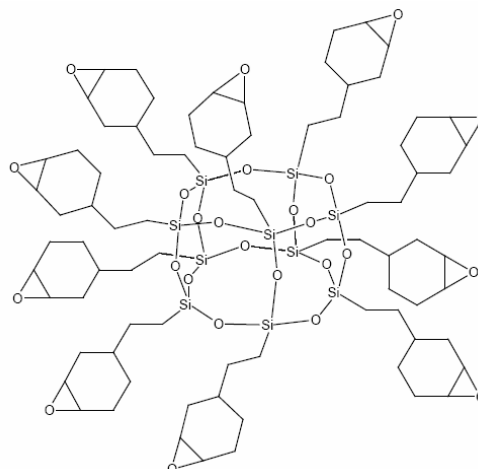
Figure 1: Chemical structures of the resin system components used in this study: A) EPON 828 ($n=0.3$), and B) Jeffamine D400 ($n=5.6$).

We investigated Trisilanophenyl-POSS (Figure 2a) as a baseline nano-scale filler for the resin. This POSS was not expected to covalently bond with the resin system. We also procured samples of Epoxy Cyclohexyl POSS (Figure 2b), comprised of a POSS core with ten pendant cyclohexyl epoxy groups. These epoxy groups were expected to be reactive with amine ($-NH_2$) group of Jeffamine D-400, and thus the POSS was expected covalently bonded with the matrix during the cure cycle. However, cyclohexyl epoxy is known to be less reactive than epoxy groups attached to DGEBA, thus, it was not known to what extent the POSS would bond with the matrix.



R = phenyl

A



B

Figure 2: Chemical structure of A) Trisilanolphenyl-POSS structure, and B) Epoxy Cyclohexyl POSS.

Trisilanolphenyl-POSS is a white powder (Figure 3A). A first sample of EpoxycyclohexylPOSS was ordered from Hybrid Plastics in early 2005 (Figure 3B). Several unsuccessful attempts were made to dissolve this into a solvent or into the epoxy. We attributed this problem to premature curing of the EpoxycyclohexylPOSS. All POSS samples were stored in a refrigerator thereafter. A second sample of EpoxycyclohexylPOSS obtained from Hybrid Plastics was in the form of a highly viscous solution in THF (3%). This sample was not very stable and began to dry out, so we diluted it to 50% with THF (Figure 3C). The diluted sample was rheologically stable throughout the program.



A



B



C

Figure 3: A) Trisilanolphenyl-POSS, B) first sample of Epoxy Cyclohexyl POSS (neat), C) second sample of Epoxy Cyclohexyl POSS dissolved in tetrahydrofuran (THF) at 50wt%.

In addition, we prepared a few samples with SNA layered silicate from Southern Clay Products. The SNA was treated via exchange chemistry with octadecylammonium chloride through proprietary procedure to enhance exfoliation.

2.2. Sample fabrication

Resin preparation: Hybrid Plastics advised us that the Trisilanolphenyl-POSS and Epoxy Cyclohexyl POSS were fully soluble in epoxy resin. However, we experienced a difficult time achieving full solubility. We had to heat EPON 828 to 100°C, add the POSS, and mix by hand every 5 minutes for about an hour while keeping the resin at 100°C. The curing agent Jeffamine D-400 was not added at this time because it would cure the resin. POSS was added to the preheated EPON 828 at a level of 5 wt% or 10 wt% relative to the final epoxy/amine weight. After the POSS was solubilized, the resin was cooled, and Jeffamine D-400 was added and mixed by hand. The sample was then degassed in a vacuum oven at room temperature for 10 minutes. The temperature at which the Jeffamine was added and sample degassed was 70°C for Trisilanolphenyl-POSS and 20°C for Epoxy Cyclohexyl POSS. There was a small amount of undissolved POSS in the 10% sample, which dropped to the bottom. Therefore, we suspect the solubility limit is between 5 and 10 wt%

Bulk samples

Bulk samples were created by pouring degassed nanocomposite resin into the “plug” fixtures provided by AFRL (Figure 4a) or small open top molds to create samples for DMA testing. The cure cycle was 80°C for 2 hrs. and 125°C for 3 hrs. We used a stereomicroscope and gratical to help quantify the needle spacing and degree of alignment in the plug samples (Figure 4b).

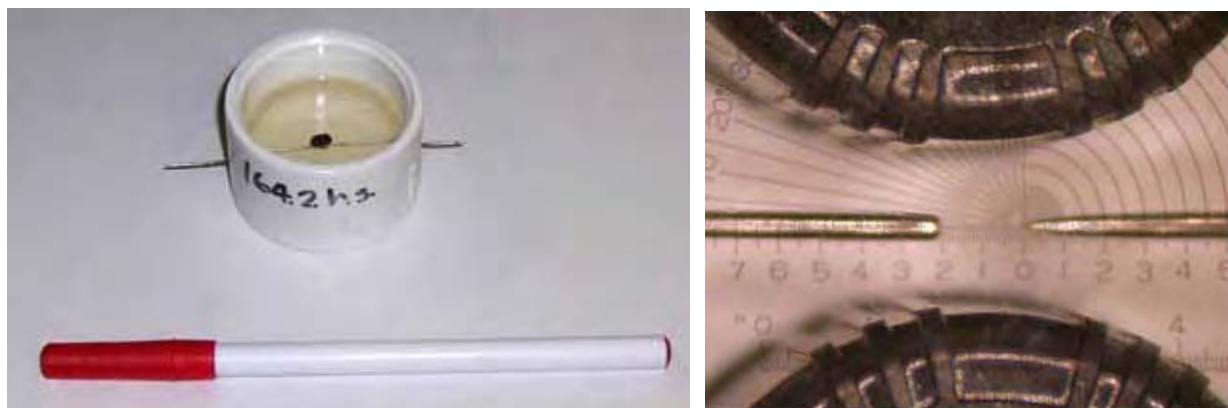


Figure 4: A) bulk plug sample, B) close-up image of needle spacing and alignment in typical plug sample.

Film forming:

A significant effort was required to develop a method of forming low-void, high quality thin films of the correct dimensions (0.2 mm thick) for electrical testing at AFRL [16,17]. We initially attempted to simply film-cast the epoxy mixture on glass and Teflon paper. The resin

would wet the surface if it had not been coated with a release agent, but then the film could not be removed. If the glass was coated, then the epoxy would not wet the surface and therefore form beads. Also bubbles would form on the surface even after being degassed in a vacuum oven. Trials were then made in silicone molds compressed with metal clamps, but they tended to bow and the films were not sufficiently thin or consistent in dimensions. Next we tried using metal or glass molds, but the epoxy adhered strongly to these surfaces and could not be removed. We evaluated three mold release agents from Rudolph Bros for their performance on glass. It was found that the release agents worked much better when they were applied in a series of thin layers and heat treated at about 60-70°C. The 700 NC worked the best so it was used for all films on glass.

To achieve a more consistent thickness in the film, a new method was developed. First, a sheet of glass was prepared with several coats of release agent, as described above. Tape was applied to the edges of the glass plate for a thickness guide. A mating surface comprised of Teflon was clamped to the glass plate to form a narrow but wide cavity between the plates. A 16-gauge needle was speared through the Teflon to reach the empty cavity on two opposite ends of the set-up. The degassed resin was injected through one of the needles and a water aspirator was used to create a vacuum suction and draw the resin across the plate. This method worked well. The Teflon/glass plates were easy to separate and films were fairly easy to remove. However, the film picked up the texture of the Teflon plate. The thickness was very uniform and there were no bubbles. The films were allowed to partially cure in the plates at room temperature for 5 days before opening.

Two new 6 inch x 6 inch Pyrex plates were fabricated by the glassblower. Two ports were added to the upper glass plate, in replacement of the upper Teflon plate (Figure 5A). At first these plates were very difficult to open and prone to chipping. Therefore the corners were ground to allow for insertion of a razor blade to aid in opening the plates. 0.2-mm-thick Kapton tape was used to maintain a constant thickness gap, as well as to cover and protect the more delicate corners. Several coats of release agent were applied to the plates.

The plates were clamped together tightly and backed with rigid foam (Figure 5B). A vacuum was applied to one port using a water aspirator. The resin was added to one spout by a syringe and drawn across the plates to the other port. After bubbles were removed via liquid flow, the vacuum was stopped and the fixture was allowed to stand overnight at room temperature. The resin was allowed to reach a state of gelation overnight; it was then removed from the fixture the next day by peeling it off the plate. The flexible film was smoothed out on a plate and post cured in an oven at 80°C for 2 hrs. and 125°C for 3 hrs. Films of uniform thickness were achieved. This method worked very well. The films were very consistent in thickness and were easy to remove from the glass. Films were then made adding 5 and 10% POSS, which flowed just as well as the neat resin samples.

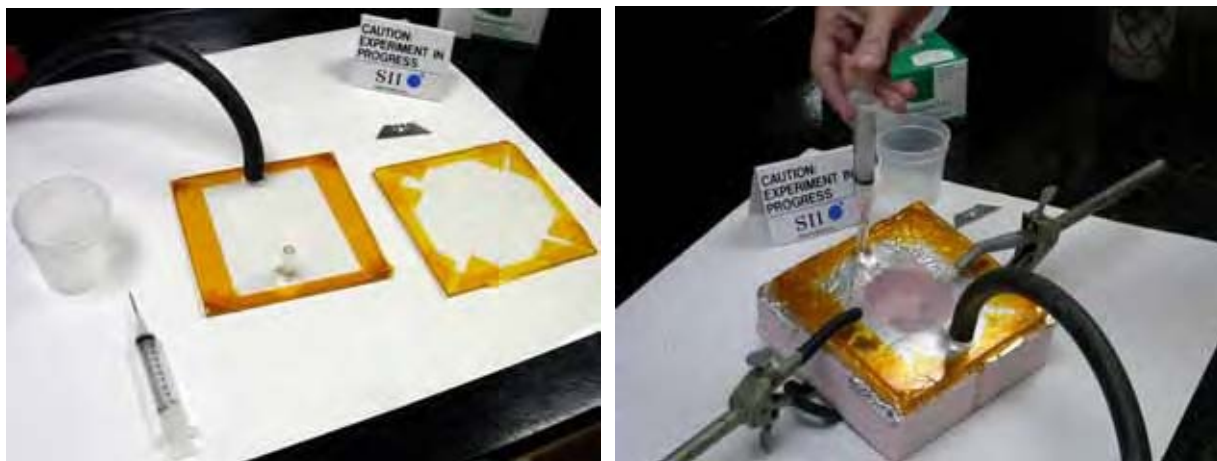


Figure 5: *thin film fixture, A) disassembled, B) assembled and showing resin injection.*

2.3 Analysis

The microstructure of the nanocomposites was examined by analyzing the fracture surface of thin film samples. A film was soaked in liquid nitrogen, then removed and immediately fractured. The specimens were then sputter-coated with gold and examined in a Hitachi S-4800 High Resolution Scanning Electron Microscope (HRSEM) at 15 keV.

The glass transition of the nanocomposites was measured with a TA Instruments 2980 Dynamic Mechanical Analyzer (DMA). Samples (approximately 50 mm x 8 mm x 3 mm) were tested in 3-point bending at a heating rate of 2°C/min and frequency 1 Hz.

Viscosity was measured with a TA Instruments AR-2000 parallel plate rheometer in dynamic mode. Test conditions were 1% strain, 1 Hz, 1 mm gap, and 5°C/min heating rate.

Several thin film and bulk samples were sent to AFRL for electrical testing.

3. Results & Discussion

A list of samples produced during the program are given in Table 1.

Sample Type	Filler	Filler Concentration (by weight)	# samples
Bulk / plug	none	--	5
	Trisilanolphenyl-POSS / THF	10%	1
	Trisilanolphenyl-POSS	5%	1
	Layered silicate (nanoclay)	3.5%	1
	none	--	4
	Trisilanolphenyl-POSS	5%	4
	Layered silicate (nanoclay)	3.5%	1
Films	none	--	3-4 ^a
	Trisilanolphenyl-POSS	5%	2 ^a
	Trisilanolphenyl-POSS	10%	1 ^a
	Epoxy Cyclohexyl POSS	5%	1 ^a
	Epoxy Cyclohexyl POSS	10%	1 ^a

^a Each film sample yielded at least 4 test specimens for AFRL

The quality of POSS dispersion was judged first by visual analysis of the samples. Optical clarity indicates good dispersion at least down the micron range. Examples of *poorly dispersed* (prior to process refinements) and *well dispersed* (after process refinements) samples are given in Figure 6A. Samples containing up to 10 wt% Epoxy Cyclohexyl POSS were sufficiently low in viscosity to form good films, and their optical clarity was very good to excellent (Figure 6B). The optical clarity was better at lower loadings of POSS. In hindsight, loadings of 10 wt% are very concentrated relative to what Hybrid Plastics generally recommends (~ 1 wt%). Thus, it is promising that we achieved good results at high loadings.

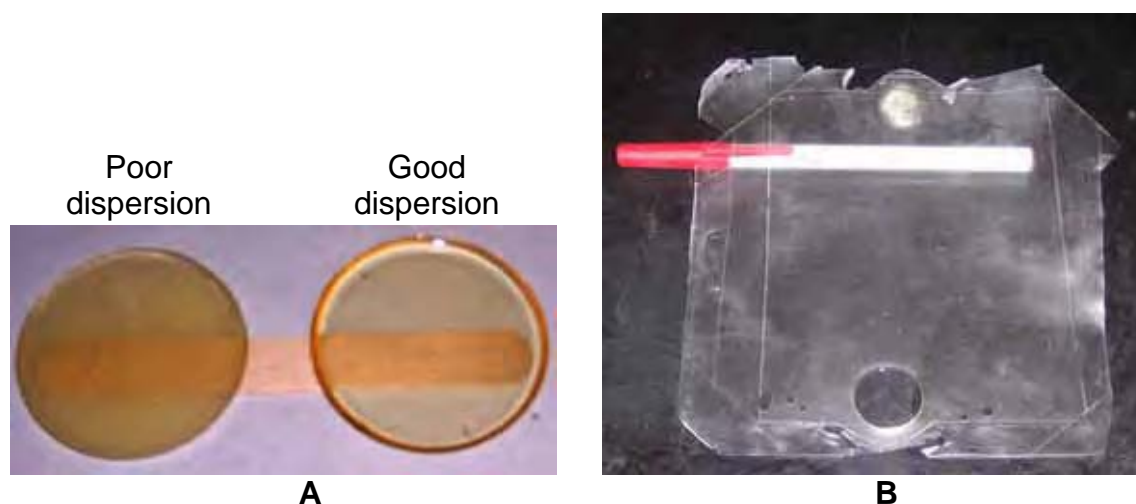


Figure 6: A) examples of poorly dispersed and well dispersed POSS-epoxy nanocomposites, and B) 10% POSS-epoxy nanocomposite film, showing excellent transparency.

HRSEM analysis indicated that there were still some agglomerated POSS particles present in the samples (Figure 7). However, the agglomerates are less than 500 nm in diameter. The amount of fully dispersed POSS is difficult or impossible to determine by HRSEM analysis.

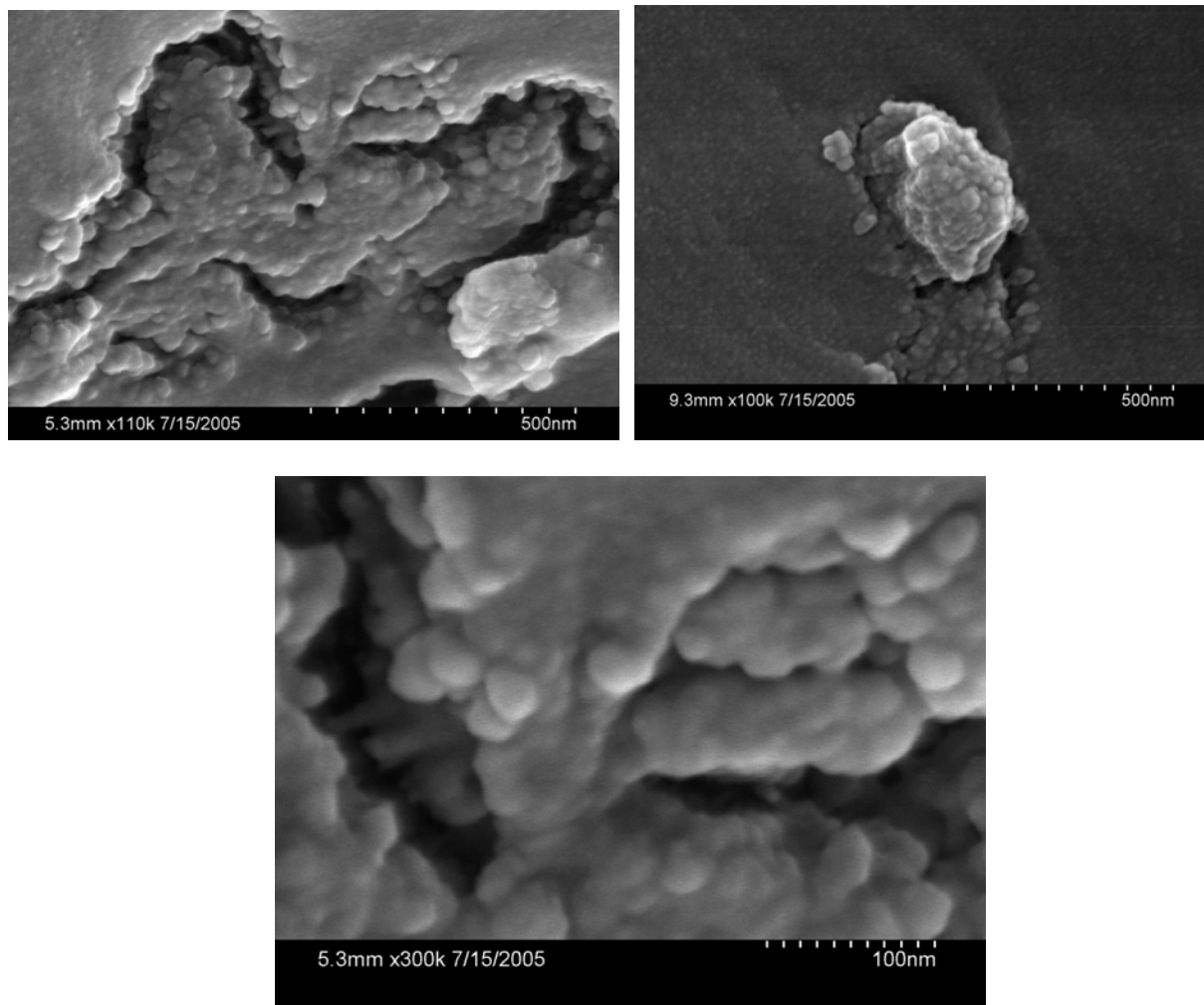


Figure 7: HRSEM images of fracture surface of POSS nanocomposite.

A typical DMA result is given in Figure 8. The results summarized in Table 1 show a slight T_g improvement with addition of Trisilanol POSS, but no improvement with Epoxy Cyclohexyl POSS. It is generally known that T_g in thermoset polymers is heavily influenced by network formation and cross link density. The epoxy groups of the Epoxy Cyclohexyl POSS add to the overall epoxide content of the resin system, thereby shifting the mixture off of stoichiometric balance. This would result in a lowering of the T_g . However, it is possible that some fraction or all of the cyclohexyl epoxy groups did not react at the given cure cycle temperatures. In either case, the Epoxy Cyclohexyl POSS did not negatively affect the T_g to any significant level compared to the base resin. The storage modulus (E') at room temperature was increased with

all POSS samples compared to baseline resin. This is not surprising, given that properties in the glassy state are generally not heavily dependent on cross link density, while factors such as particle loading become important. The increased modulus values for the nanocomposites thus reflect the presence of well dispersed POSS loading.

The rheometer results (Figure 9) show that the addition of POSS raises the viscosity of the resin, but only by a small amount. Furthermore, heating can be used to adjust the viscosity.

Electrical test results are summarized by AFRL elsewhere [16,17]

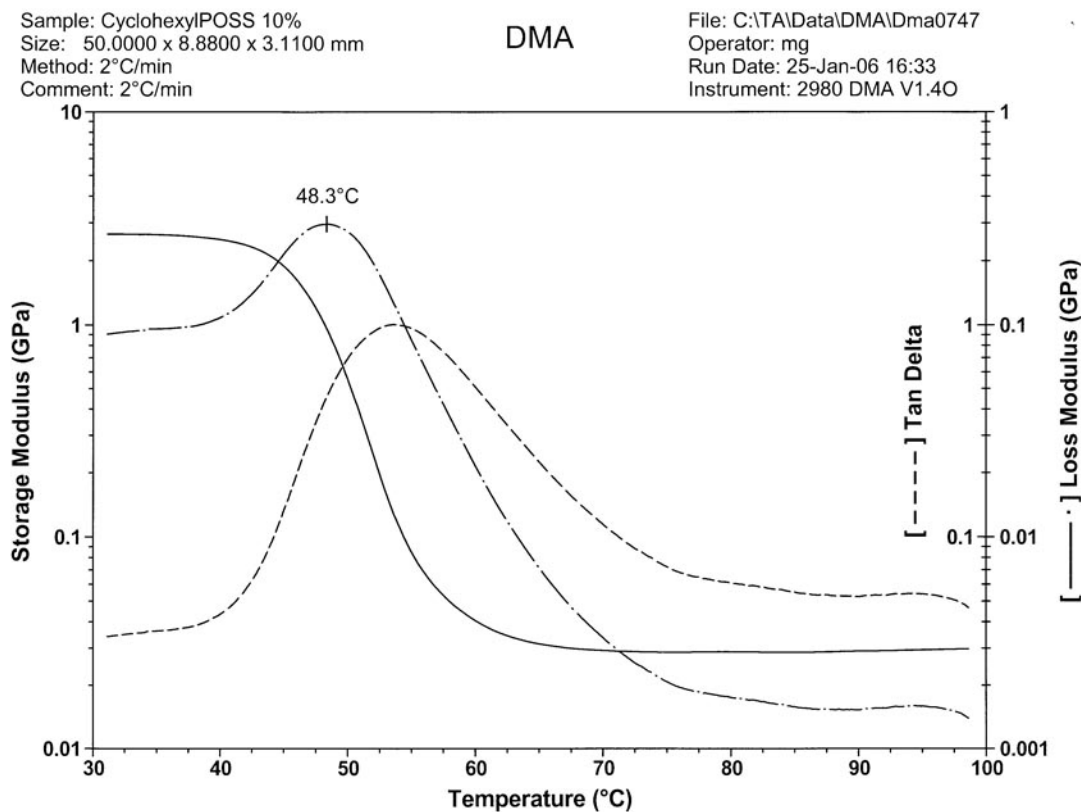


Figure 8: Typical DMA result, showing T_g taken from the peak of the Loss Modulus curve for a 10% Epoxy Cyclohexyl POSS sample.

Table 1: DMA results

Sample	T_g^2 ($^{\circ}\text{C}$)	E' (GPa) ³
Base resin ¹ (no POSS)	49	2.1
EPON 828 + Epikure W (no POSS) ⁴	135	3.8
Base Resin + 5% Trisilanol POSS	53	2.5
Base Resin + 5% Epoxy Cyclohexyl POSS	49	2.8
Base Resin + 10% Epoxy Cyclohexyl POSS	48	2.8
Base Resin + 3.5% nanoclay	51	2.2

¹ EPON 828, Jeffamine D-400, stoichiometric mix, standard cure cycle (2 hrs @ 80 $^{\circ}\text{C}$, 3 hrs @ 125 $^{\circ}\text{C}$)

² taken from peak of the loss modulus (E'') curve

³ room temperature value

⁴ Epikure W is an aromatic diamine curing agent; this sample was cured for 2 hrs @ 177 $^{\circ}\text{C}$

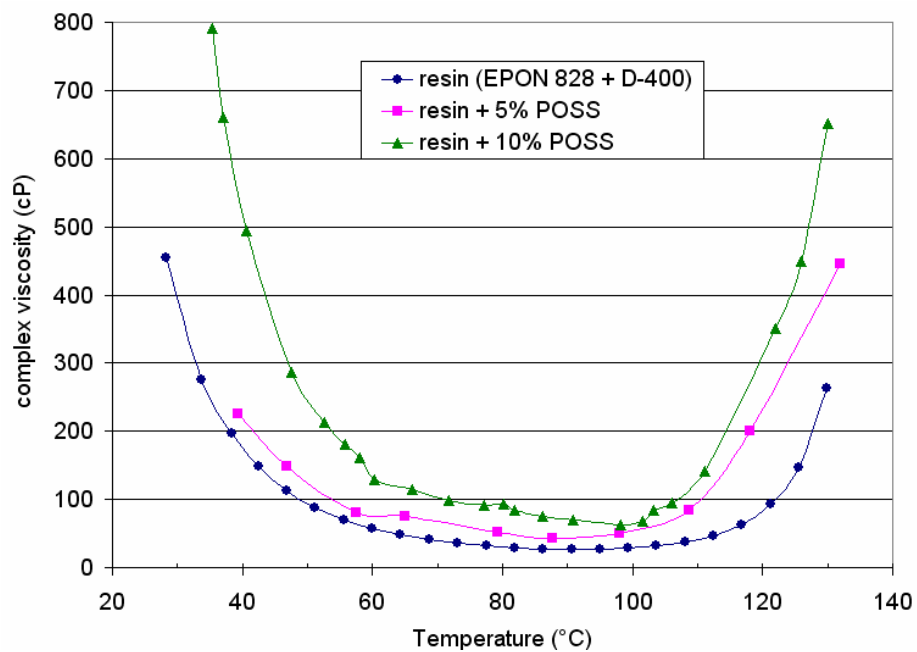


Figure 9: Parallel plate rheometer results for EPON 828 + Jeffamine D-400 + Trisilanol POSS.

4. Conclusions

A procedure was developed to incorporate POSS nanoparticles into a common epoxy/amine resin system at loadings over 5 wt%, where we believe the solubility limit to be between 5 and 10 wt%. Dispersion of the particles was excellent in regards to optical clarity. Closer examination with HRSEM indicated that dispersion down to the 1-10 nm level was not accomplished for all the particles – agglomerates of up to 500 nm could be detected. It was not clear if the Epoxy Cyclohexyl POSS particles bonded with the matrix. There was no T_g improvement compared to unfilled resin, and storage modulus improvements was only slightly greater than the Trisilanol POSS samples. Overall the bulk and thin film samples provided to AFRL for electrical breakdown testing were of high quality, although further dispersion quality improvements are possible. Additional analysis of the extent of reaction between Epoxy Cyclohexyl POSS and the epoxy resin is also a potential area of further research.

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